NATIONAL BUREAU OF STANDARDS REPORT

6109

Progress Report

on

A SETTING TIME TEST FOR DENTAL AMALGAM

by

Duane F. Taylor
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U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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A SETTING TIME TEST FOR DENTAL AMALGAM

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ABSTRACT

A setting time test has been developed for dental amalgam based upon the shear strength of freshly condensed amalgam. The load required to punch a circular hole in the amalgam is measured at various times after condensation. It is found that the time at which the required load reaches 25.5 lbs, corresponds closely with the clinical limit of carvability as determined by a group of dentists.

A SETTING TIME TEST FOR DENTAL AMALGAM

1. INTRODUCTION

From the point of view of clinical application, one of the most important characteristics of dental amalgam is the rate at which it hardens or sets. An amalgam which sets too rapidly or too slowly is a great inconvenience to the dentist and can be the direct source of faulty restorations. The clinical importance of setting time has been recognized for well over 50 years. G. V. Black reported as early as 1895 that some of the factors influencing it were well known to the manufacturers of that time [1]. Most investigators since then have been aware of the problem and, in many cases, have emphasized it in their publications [1-6]. Nearly all amalgam research, however, has been directed toward other properties such as setting expansion, strength and flow, and almost nothing has appeared in the literature about the objective measurement of setting time.

The lack of information about the setting time of commercial amalgam alloys has necessitated a trial and error search on the part of the dentist for an alloy that suits his particular technique or preferences. The useful range of setting times appears to be well covered by the products currently on the market, although the large number of alloys may, in itself, add to the dentists' difficulty in selecting a suitable one.

In the case of the Armed Forces dental services, or in any case where the dentist's choice of products is restricted, difficulties with setting time can become more critical, at times making an otherwise satisfactory alloy practically unusable. The development of a suitable objective test for setting time should help solve these difficulties.

2. THEORY OF SETTING

The mechanism by which amalgams harden is not completely understood but it is characterized by gradual and continual physical property changes. These changes appear to follow a definite sequence, a variation in technique that accelerates one stage of the setting process also accelerating the others. However, because of the continuous nature of the physical property changes, any "setting time" test must involve the selection of a somewhat arbitrary limiting value for the measured property. Also, if the results are to have much practical meaning, the physical property measured must show good correlation with some clinically significant property of the amalgam. The property normally used as a measure of the setting time of other dental materials is indentation or penetration resistance. While consistent results can be obtained for amalgam using such methods, and they have been used by others [7] to study the effect of variation of particle size and shape, the present authors were unable to obtain correlation of indentation tests with clinical estimates of setting-time. The clinical characteristic chosen for comparison was the "limit

of carvability" - that time at which the dentist feels that further carving is apt to damage the restoration.

Freshly condensed amalgam may be considered as resembling a mass of wet sand, the remnants of the original alloy particles being packed into close contact with each other and the intervening spaces filled with a solution of the alloy in mercury. The amalgram differs from the sand analogy mainly in the rapid hardening of the interparticle matrix that occurs as the matrix continues to alloy with the particles which it surrounds. It is felt that carving is essentially a shearing process dependent upon the properties of the matrix materials while indentation is in large part a function of the shape, strength and packing behavior of the particles. On this basis, the poor correlation of indentation tests with clinical observation can be explained, since a large and variable amount of the resistance to indentation would be the result of a "wet sand effect" due to packing of the particles rather than to any setting reaction.

3. EXPERIMENTAL PROCEDURE

3.1 Material

The four amalgam alloys chosen for use in this study are listed in Table 1. All are certified by the manufacturers to meet the requirements of American Dental Association Specification No. 1. for Dental Amalgam Alloy. They were selected on the

basis of preliminary tests as providing a representative range of setting times and particle sizes.

Table 1

Brand	Manufacturer	Lot No.
Silver Crown Slow	General Refineries, Inc.	2110
True Dentalloy A Cut	S. S. White Dental Mfg. Co.	02652303
20th Century Regular	L. D. Caulk Co.	7J54R
20 Century Fine Cut	L. D. Caulk Co.	22H55F

Various lots of mercury were used, some being certified brands of dental mercury and others being from chemical supplies. All lots, however, met the requirements of American Dental Association Specification No. 6, for Dental Mercury, indicating a non-volatile residue of less than 0.02 percent.

3.2 Equipment.

All weighings of alloy and mercury were made on a torsion balance to the nearest 5 mg. Bakelite amalgamator capsules with bakelite pestles (U.S. Armed Services medical

supply No. 6250-500-5150) were used with a Crescent "Wig-l-Bug" Amalgamator. The normal operating speed of this particular amalgamator when operated with a loaded capsule was determined as being 3330 cycles per minute by means of a General Radio "Strobotac" type 631-b.

Duration of trituration was measured by the automatic timer on the amalgamator to 0.2 second. All other time measurements were made with stop watches.

All load applications were made by means of a Tinius-Olsen Universal Tester of the pendulum type. A 1000 lb range graduated to 0.5 lb. was used for condensing specimens and a 100 lb. range graduated to 0.05 lb. was used for testing.

A four piece specimen mold of the type shown in Figures 1 and 2 was prepared of tool steel. All pieces were machined, hardened and then ground to fit. The four pieces are; an outer block with a cylindrical hole through it; two plungers with flat heads, one used as a spacer to fix the size of the specimen cavity while amalgam is being placed in the mold, and the other used during condensation to apply pressure to the top surface of the specimen; and a headless plunger with a groove that serves to collect the mercury expressed as the packing load is applied. The method of assembling these parts for both loading and pressing is shown in Figure 2.

The absolute dimensions of the mold parts are of much less importance than their relative fit and the perpendicularity of the plunger ends to their longitudinal axis. The plungers were made to fit the hole in the outer block as closely as possible and still permit routine hand assembly. A flat was ground on the lateral surface of the spacing plunger to prevent the trapping of air during assembly. The one exception to the tight fit was the flange on the headless plunger separating the mercury well from the specimen. This clearance was intentionally increased 0.005 inch to facilitate the expression of mercury. The dimensions of the outer block are approximately 1 by 1 by 1 1/2 inches, the hole diameter approximately 0.70 inches. The combined length of the headless plunger and the spacing plunger were such as to leave a specimen cavity 0.040 inches deep. The perpendicularity of the end faces of the headless and pressing plungers to their axis was such that the maximum observed differences between the thickest and thinnest edge areas of specimens made in this mold was 0.0004 inch. Normal variation is somewhat less than this. This uniformity is important since nonparallelism of these opposing faces during loading may result in displacement of the excess mercury from one portion of the specimen to another rather than complete expression. Such specimens, uniform in neither composition nor dimensions, yield erratic test results.

The punch used for determining setting time is illustrated in Figures3 and 4. The two essential parts, the punch itself and the matching female die, are mounted in a heavy alignment block so as to provide a bearing of sufficient length and stability to assure the proper alignment of the punch and die. All parts are of tool steel, machined,hardened, and ground to fit. The fit on the punch is such that it will settle slowly under its own weight when lubricated with instrument oil. The diameter of the punch is 0.250 inch and the diameter of the aligning bearing surface is 0.010 inch larger than the molded specimen. The female die is made as a separate piece and is slightly shorter than the lower hole in the alignment block base. This arrangement forms a shallow cavity over the die which is used for positioning the specimen. Both the punch and die may be removed easily so that they may be reconditioned on a surface grinder if use rounds the shearing edges. The total clearance (difference in diameter) between the punch and die is 0.0010 ± .0001 inch and, when in place, they are held concentric within 0.0001 inch.

A limited selection of carvers was made available to the dentist's participating in the carving tests. Each dentist was permitted his choice of the No. 1, 2, and 3, 105° Frahm Carvers (right, left, and straight).

3.3 Specimen Preparation

All specimen preparation and testing was done in a controlled temperature room maintained at 72°F and 55% R. H.

Specimens of the same type were used for both the carving and shear tests. These specimens were prepared in the following manner:

Three amalgamator capsules were used for the trituration of the amalgam for each specimen. Each capsule was loaded with 0.400 ± 0.002 grams of alloy and sufficient mercury to satisfy the manufacturer's prescribed mercury-alloy ratio. These loads were triturated in sequence for seven seconds each on the amalgamator, twenty-five to thirty seconds being required for the operation. The start-of-mix for measurement of the age of specimen was taken as the start of trituration of the second capsule load of amalgam. At the end of trituration, the three portions of amalgam were combined and placed in the specimen cavity of the mold as assembled for loading (see Figure 2). There the amalgam was distributed by a troweling motion with a small glass plate (2 x 2 inches slide glass). When the mold was filled, the straight edge of the glass plate was used to scrape off the excess amalgam flush with the top surface of the mold block, producing a specimen of wet amalgam 0.040 inch thick. Throughout this procedure, every effort was made to keep the manipulation of the alloy to a minimum.

The mold was then assembled for pressing, and load application was begun 2-1/2 minutes after the start of mix. Load was applied with the testing machine at a rate equivalent to 55 psi/sec until a pressure equal to 2000 psi was reached. After 4.5 minutes from the start of mix, the load was removed at a rate of approximately 400 psi/sec. The specimen was removed from the mold and stored at 72°F and 55% R. H. until tested.

3.4 Carving Test Procedure

The procedure employed in the carving test was designed to avoid any influence upon the participating dentists' estimates of the time at which the amalgam set. The following routine was observed throughout the tests:

The dentist performing the test was permitted his choice of the three carvers.

He then received the amalgam specimen without any information as to when it was expected to set. He also received, face down, a stopwatch measuring the age of the specimen. He was instructed to carve the amalgam as he saw fit until it hardened to a condition where he felt that further carving in the mouth might risk fracture of a restoration. He was then to stop the watch.

The specimens were given to the dentists at times ranging from one to four minutes before the anticipated time of set in order to avoid the bias that might occur if the participants came

to expect set to occur some fixed interval after they started carving. As further insurance against bias, the brand of alloy tested and the age of the specimen were also concealed from the dentist.

Eight dentists participated in the carving tests, with at least four dentists carving each alloy. The number of specimens carved per alloy varied from nine to twenty. The results of these tests are presented in Table 2.

3.5 Test Procedure

Specimens, prepared in the standard manner and identical to those employed for the carving test, were used for the punch test. Approximately one minute before the time of test, the specimen was positioned in the lower die and the entire punch assembly was placed in the testing machine. The machine was then adjusted to contact the punch without applying load. At the time selected for testing, the machine was started and load applied at a rate of 1.58 pounds per second. The load required to produce fracture was recorded to the nearest 0.1 pound. A series of specimens were run at each of several times for each alloy, and the curve showing the progress of the setting reaction was derived from them. Figure 5 shows a plot of the observations on Alloy B. The plus marks indicate individual readings and the circles averages of all readings at a given specimen age. The results of all of the punch tests are presented in Table 3.

4. DISCUSSION OF RESULTS

Examination of Figure 5 shows that a straight line is a good fit to the data within the range of time investigated. Similar plots of the results for the other alloys show this to be a common characteristic. Combination of these results with those from the carving tests permit the estimate of the shear strength (measured in terms of punch load) of the amalgam at the limit of carvability. This estimation is indicated by the dashed lines in Figure 5, the vertical dashed line being drawn at the limit of carvability (see Table 2) and the horizontal dashed line showing the derived shear strength.

If the two tests actually measure the same property or group of properties, the same punch load should be required for shearing each alloy tested at the time of clinical set as determined by the carving test. Figure 6 shows the average shear strength lines for the four alloys and derivation of the characteristic punch load at the carvability limit for each alloy. There is a readily observed tendency for these punch loads to cluster about a constant value and therefore the two tests presumably do measure the same property. The punch test, then, can be used to provide valid estimates of clinical carving time.

The average of the individual values for the four alloys gives a punch load of 25.5 pounds at the time of set. This is equivalent to a shear stress of approximately 1150 psi. This average value may, in turn, be used to obtain an estimate of the setting time of each individual alloy. Table 4 compares the setting times so estimated with the carving times for each alloy. The maximum differences between the estimates are the same or smaller than the uncertainties by either method.

The general agreement between the shear test results and the carving tests over a wide range of setting times indicates that both tests measure the same property. The shear test therefore should be a valid objective test for the clinical limit of carvability.

5. SUMMARY

A test is described to measure the setting time of dental amalgam. The test measures the load required to punch a 0.250 inch diameter hole in an amalgam disc approximately 0.030 inch thick. The time at which the required load is 25.5 pounds is found to correspond closely to the "limit of carvability" as estimated by a group of dentists for four popular alloys. This is taken as an indication that both the punch test and hand carving are measuring the same property and that, therefore, the punch test may be used validly to provide a quantitative objective measurement of clinical setting time of amalgam.

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Table 2

SETTING TIME OF AMALGAM

AS DETERMINED BY HAND CARVING

Alloy	Number of Specimens	Setting Time	σ
A	20	10.2 min	1.1 min
B	9	12.7	1.5
C	9	14.8	2.1
D	9	19.4	3.8

σ equals standard deviation

Table 3

AVERAGE SHEAR STRENGTH OF AMALGAM
WITH TIME

Alloy	Time	Punch Load	
	minutes	lbs.	σ lbs*
A	9	24.7	2.3
	12	31.5	1.3
	15	34.8	2.0
B	12	24.6	1.6
	16	28.6	2.5
	20	31.3	2.7
	24	36.1	2.8
C	11	19.4	2.3
	15	27.2	2.5
	25	40.5	3.1
D	15	21.3	1.3
	20	25.0	1.4
	25	29.3	2.9

* σ equals standard deviation

Table 4

COMPARISON OF SETTING TIMES
AS DETERMINED BY
CARVING TEST AND PUNCH TEST

Alloy	Carving Test Setting Time	σ*	Punch Test Setting Time	σ*
A	10.2 min	1.1 min	9.2 min	1.0 min
B	12.7	1.5	12.9	1.8
C	14.8	2.1	14.5	1.4
D	19.4	3.8	20.5	1.8

* σ equals standard deviation

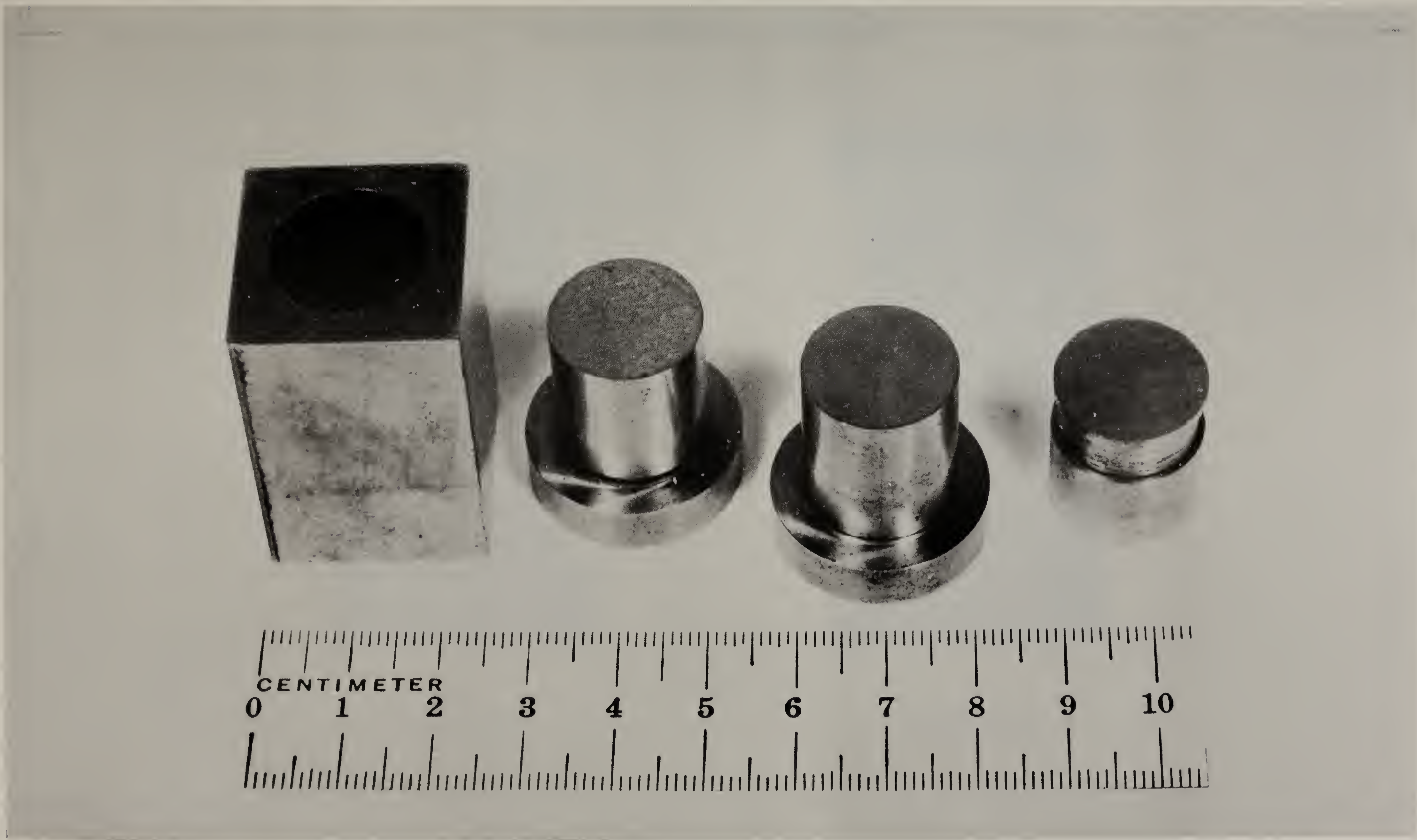


Figure 1. Specimen mold showing four pieces and approximate size.

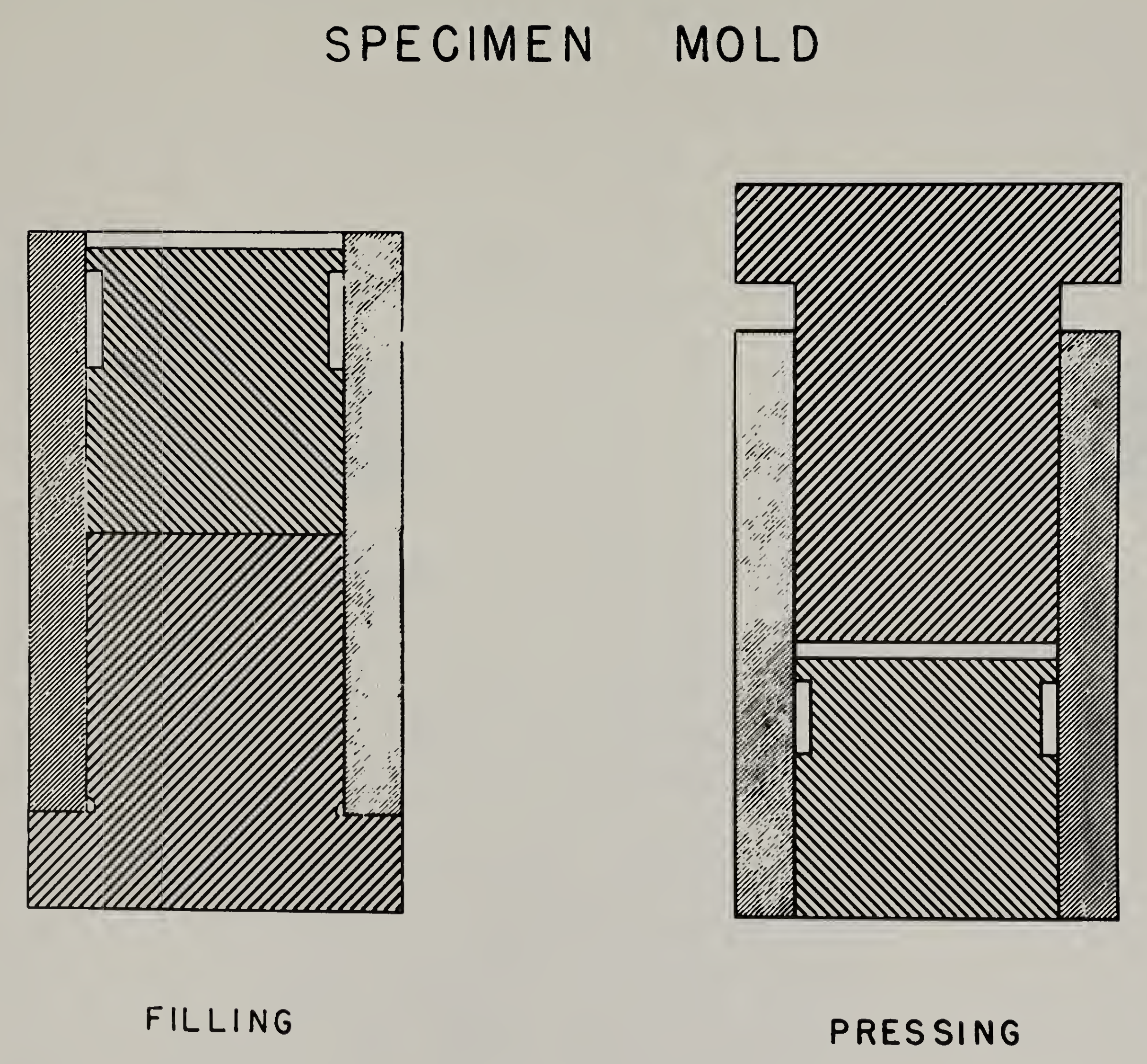


Figure 2. Schematic drawing of specimen mold showing filling and pressing positions.

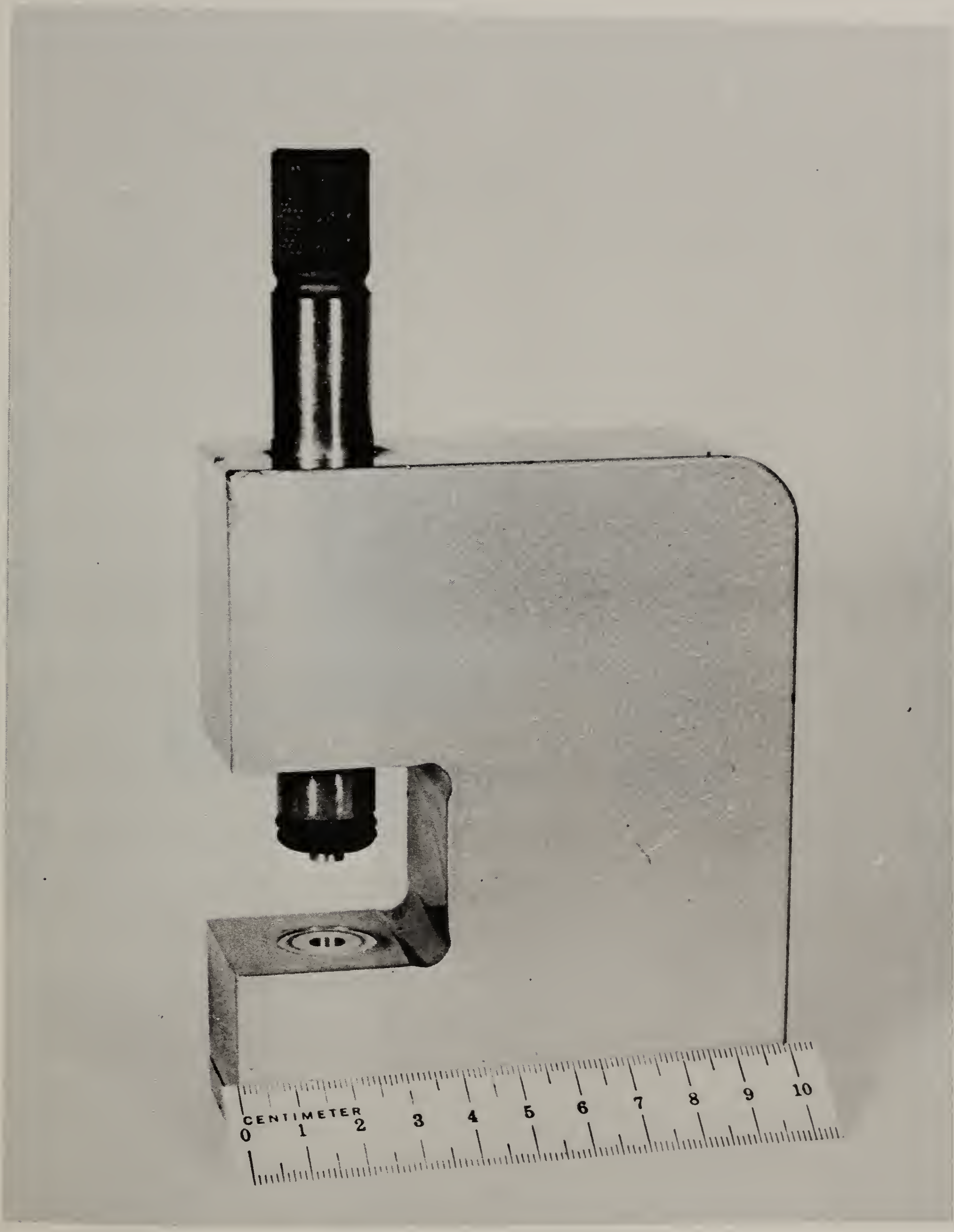


Figure 3. Setting time punch showing size in centimeters.

SETTING TIME PUNCH

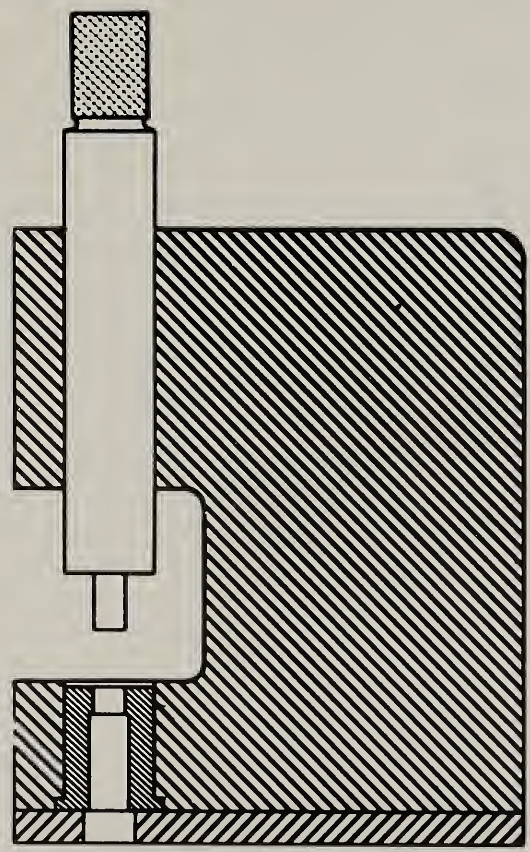


Figure 4. Schematic drawing of setting time punch showing the relative positioning of the parts.

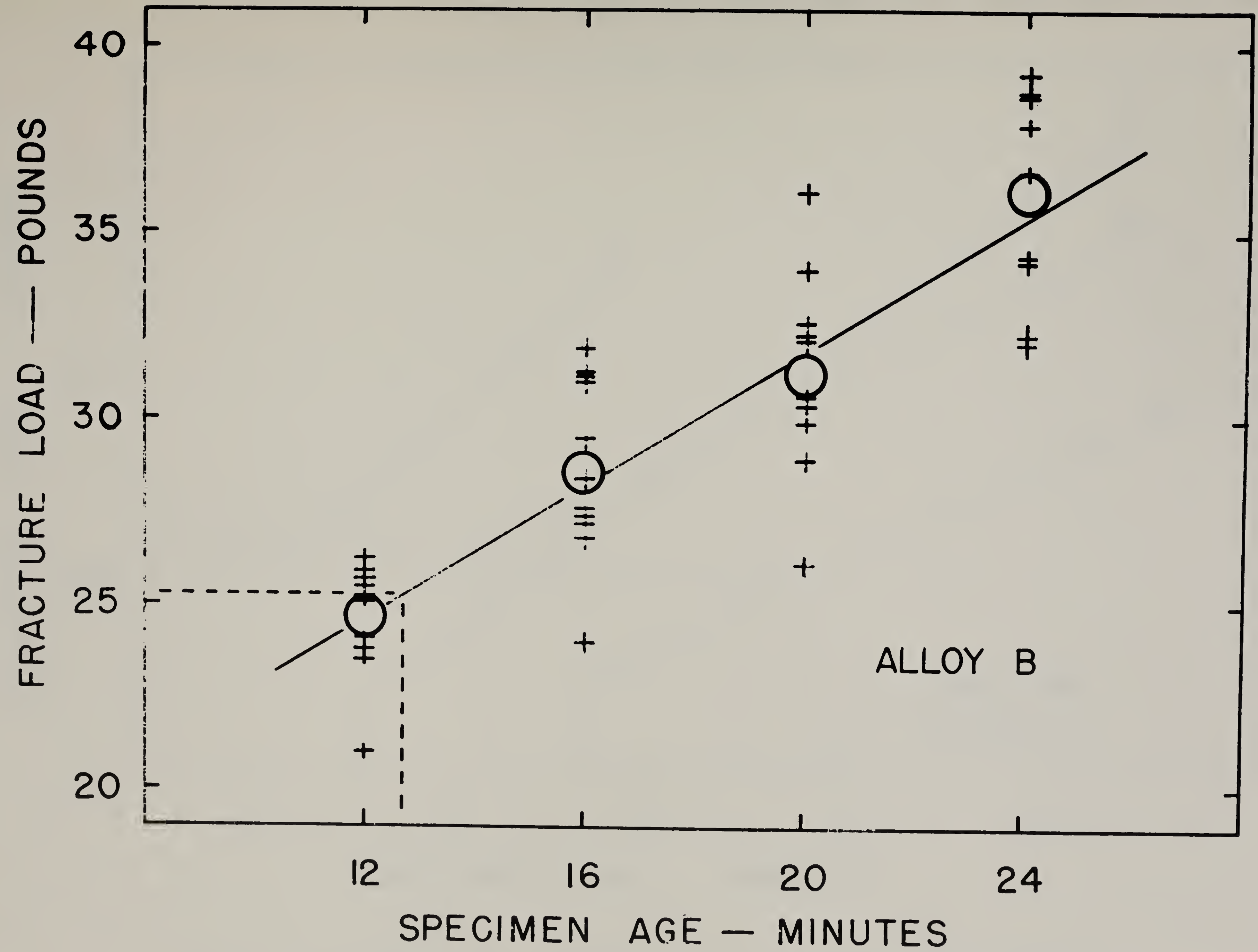


Figure 5. Plot of punch test observations for Alloy B showing linear relationship between punch shear strength and specimen age.

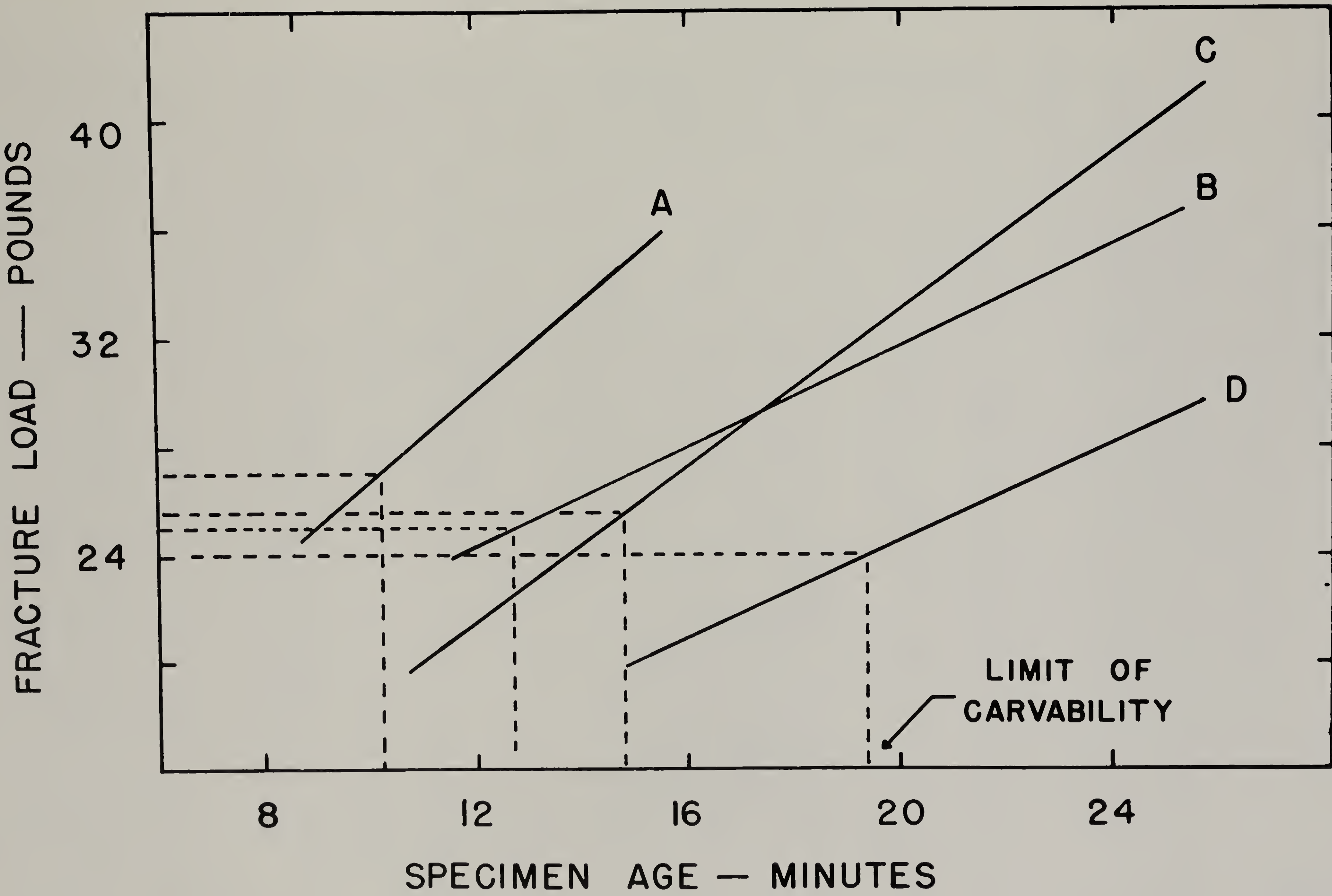
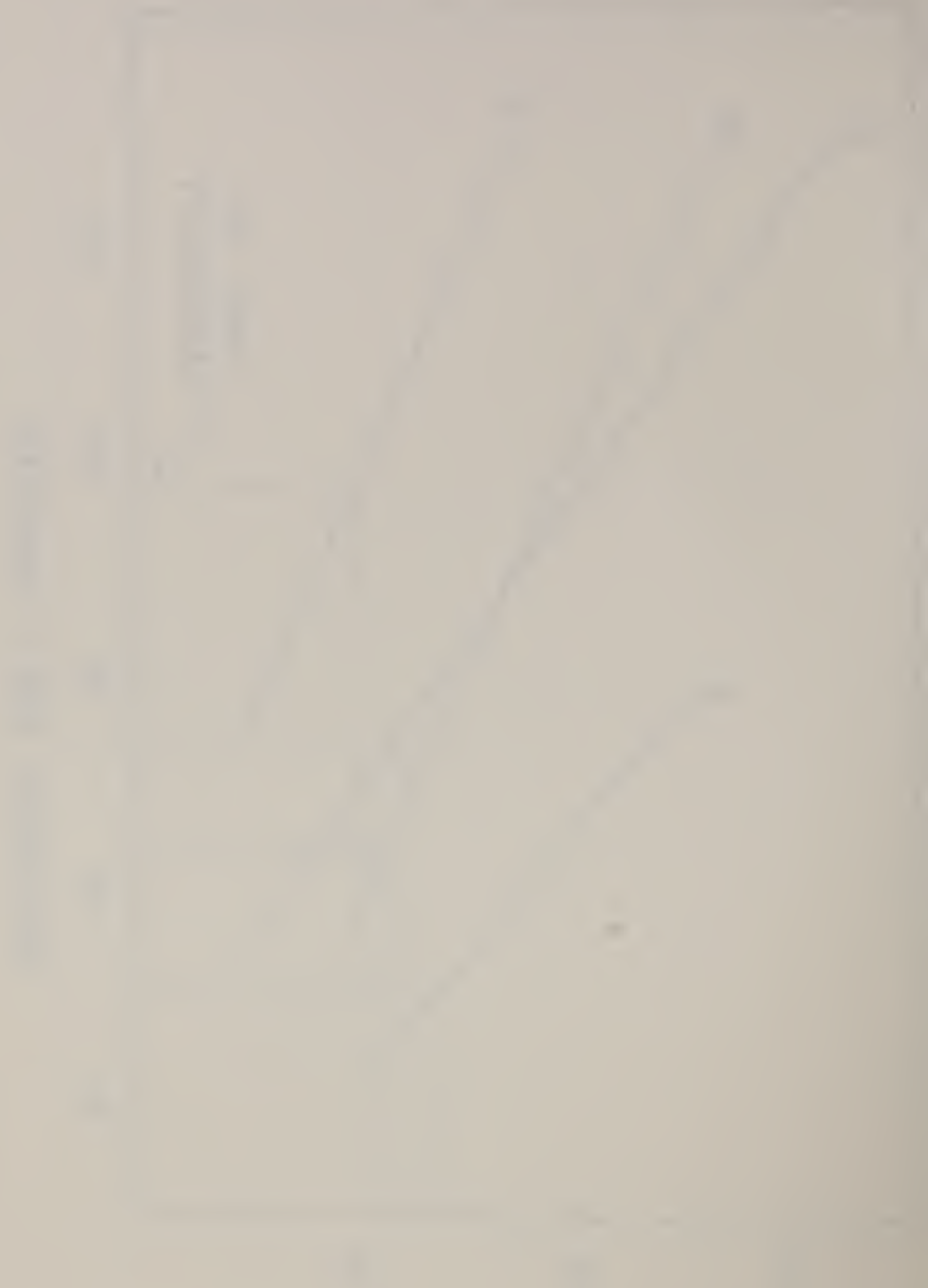


Figure 6. Average shear strength lines for four representative alloys showing derivation of characteristic punch load at carvability limit for each alloy.



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